MINERALOGICAL STUDIES OF A HIGHLY ^{17}O-DEPLETED AND AN ^{17}O-RICH PRESOLAR GRAIN FROM THE ACFER 094 METEORITE.

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Introduction: Silicate grains are the most abundant condensate around O-rich evolved stars, including red giants, supernovae (SNe) and binary systems. These grains have been identified in abundance in primitive meteorites and interplanetary dust particles [1,2]. Astronomical observations of the silicate spectroscopic features around circumstellar disks indicate that most silicates are amorphous olivine-like grains, though some sources show a large crystalline portion [3]. Fewer astronomical observations of SN and nova silicates exist, but amorphous Mg-rich grains predominate [4,5]. The laboratory analysis of presolar silicates by transmission electron microscopy (TEM) offers more details on the structure and chemistry of individual grains. These studies provide information on the physical and chemical conditions of the parent stellar atmosphere during grain condensation. Moreover, because silicates are susceptible to secondary alteration, processing events succeeding condensation can be traced. Thus far, similar microstructures have been observed for silicates that condensed in SN outflows and in the envelopes of asymptotic giant branch (AGB) stars, but not as many of the comparatively rare SN grains have been analyzed. Here we examine the mineralogies of two presolar silicate grains having different origins.

Experimental: Isotopically anomalous silicate and oxide grains were identified in a grain dispersion sample of Acfer 094 fine-grained matrix material by NanoSIMS O isotopic imaging. A ~1 pA, 100 nm Cs^+ primary ion beam was rastered over 20 μm fields of view and the O and Si isotopes, and $^{24}Mg^{16}O$ were measured. Two anomalous grains, one ^{17}O-rich and one ^{17}O-poor, were selected for further TEM analysis. No additional isotopic analyses were conducted because of the small size of these grains (~170 nm). Due to the proximity of the grains, an electron transparent cross-section containing both grains was produced by focused ion beam (FIB) milling (Fig. 1). An electron beam deposited C cap was first placed on the grains of interest to serve as markers and to protect them from beam damage. The C contrasts nicely against the Pt strap that was subsequently deposited. To ensure that each grain was located precisely, the last stages of thinning and fine-milling were performed on each grain separately. We obtained imaging, diffraction and chemical data of the presolar and neighboring grains using the JSC JEOL 2500 field-emission STEM equipped with a Noran thin window energy-dispersive X-ray (EDX) spectrometer. Quantitative elemental maps of the sample were acquired with a 4 nm incident probe whose dwell time was minimized to avoid beam damage and element loss/mobilization during mapping.

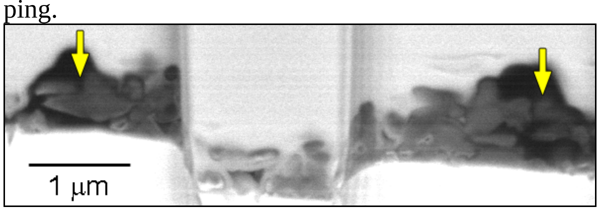

Figure 1. Backscatter electron image of the electron transparent cross-section containing presolar grains 3_13a (left arrow) and 3_13b (right arrow). The dark C cap is apparent under the Pt strap.

Results: Grain 3_13a is highly depleted in ^{17}O (δ^{17}O = -780 ± 70‰). The $^{18}O/^{16}O$ and Si isotopic ratios are normal within error. This grain has an O isotopic composition consistent with Group 3 grains [6], which are thought to derive from low-metallicity stars and supernovae. Recent O, Mg, and Si isotopic analysis of a Group 3 silicate with a less substantial ^{17}O depletion favored a SN rather than low-metallicity source [7]. A low-metallicity source for 3_13a can also be excluded because only a star of abnormally low metallicity would have the observed ^{17}O depletion and because the grain does not display a correspondingly large ^{18}O depletion. Moreover, the O and Si isotopic composition of grain 3_13a can be reproduced by SN zone mixing models [8]. The TEM analysis of grain 3_13a (Fig. 2) reveals a 130 nm amorphous silicate with a composition similar to high-Ca, Fe-rich pyroxene. The composition is non-stoichiometric and Si-rich. No compositional heterogeneities are observed and there is no evidence of crystallinity.

Grain 3_13b is enriched in ^{17}O (δ^{17}O = 660 ± 155‰) and also has a normal $^{18}O/^{16}O$ ratio, falling into the Group 1 classification. This isotopic signature is typical for grains likely originating from low-mass AGB stars. The TEM analysis shows the grain is 180×70 nm (Fig. 3) and is an amorphous Fe oxide or hydroxide grain with minor Si and S. Electron energy-loss spectroscopy measurements are planned to constrain the Fe valence and oxygen speciation. The underlying grain is a crystalline Fe-rich olivine (Fa75), while the three stacked grains to the left are amorphous Fe-bearing silicate and silica grains.

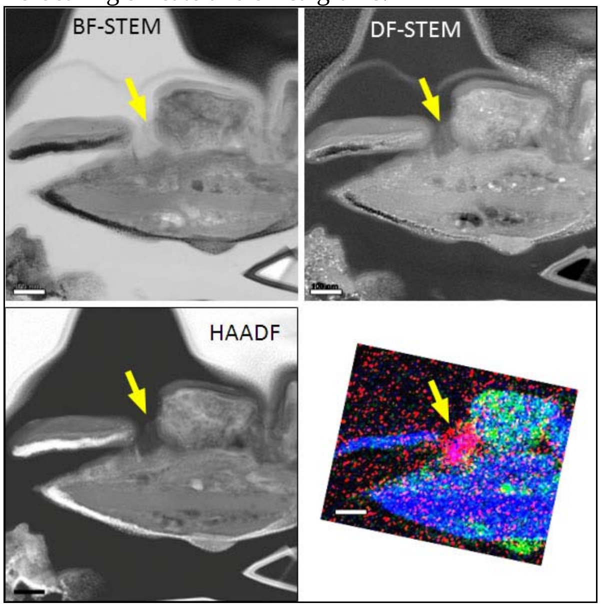

Figure 2. Bright- and dark-field STEM and HAADF images, and RGB (Ca, Fe, Si) composite elemental map of the cross-section containing 3_13a. Scale bars are 100 nm.

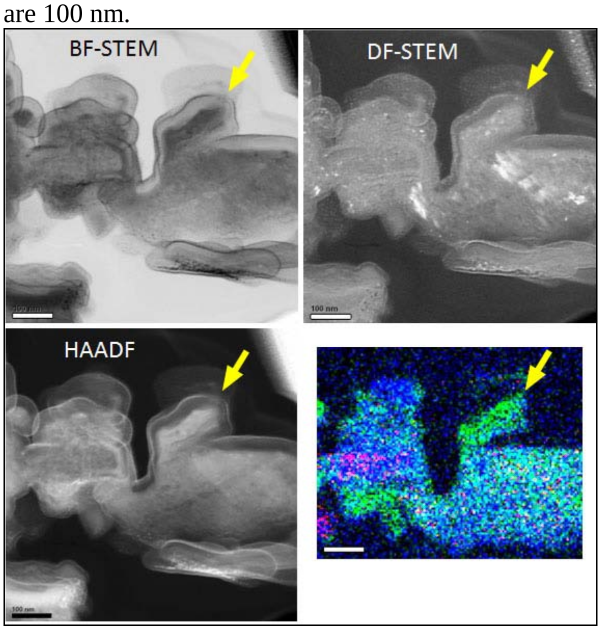

Figure 3. Bright- and dark-field STEM and HAADF images, and RGB (Mg, Fe, Si) composite elemental map of the cross-section containing 3_13b. Scale bars are 100 nm.

Discussion: Thus far, TEM analyses exist for ~35 presolar silicates of various stellar origins. These grains span a diverse range in microstructures and chemical compositions. In agreement with astronomical observations, most of the grains are amorphous and Mg-rich, though Fe contents are larger than expected. The Fe could have been acquired during grain condensation, but also during secondary alteration. The mineralogy of silicates from AGB stars and SN are similar, suggesting common physical and chemical conditions in AGB envelopes and the outer regions of SN where O-rich grains condense. The high Ca content of SN silicate 3_13a is unusual for SN condensates and suggests formation at a relatively high temperature. The Fe in this grain was either acquired during condensation or through parent body processes. Ca-rich silicates have not been observed around SN ejecta, but diopside has been fit to spectra of circumstellar regions [9] and is a predicted stable condensate in SN zones [10].

The mineralogy of about 10 presolar oxides has been analyzed by TEM (see [11] and references therein). The grains are compositionally consistent with hibonite, spinel, and corundum, and all of them are crystalline except for one Al_2O_3 [12]. Oxide 3_13b is also amorphous, though amorphization during NanoSIMS analysis cannot be ruled out especially considering the small size of the grain. One other presolar Fe oxide grain was identified that also likely originated in an AGB star [13]. Based on Auger elemental analysis, this grain is similar to wüstite (FeO), however no structural information exists.

Caution must be taken when interpreting the TEM data of the smallest grains. These grains are subjected to ion beam damage during NanoSIMS isotopic analysis and implanted Cs from NanoSIMS analysis is observed in the TEM analysis. Material may also become sputter-deposited onto the grains during isotopic analysis and FIB milling. It is not clear for the smallest samples whether they were originally amorphous or rendered amorphous through the isotopic analysis, although non-stoichiometric compositions would favor originally amorphous structures. Despite these limitations, O-rich stardust grains from a variety of sources are clearly mineralogically and chemically diverse.

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